Crystal structure of (2*E*,6*E*)-2,6-bis[2-(trifluoromethyl)benzylidene]cyclohexanone, $C_{22}H_{16}F_6O$

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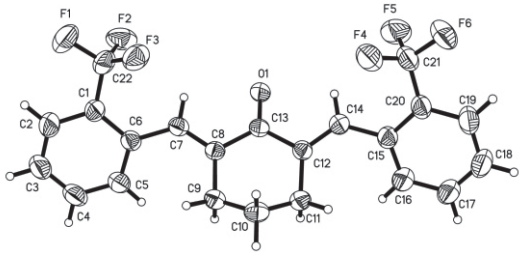

Abstract

$C_{22}H_{16}F_6O$, monoclinic, $P2_1/n$ (no. 14), a = 8.426(2) Å, b = 15.205(4) Å, c = 14.900(4) Å, β = 93.031(6)°, V = 1906.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0632, $wR_{ref}(F^2)$ = 0.1826, T = 293 K.

Table 1. Data collection and handling.

Crystal:	green prisms, size 0.147×0.212×0.276 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.27 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10678, 3547
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2398
$N(param)_{refined}$:	263
Programs:	SHELX [14]

Source of material

(2*E*,6*E*)-2,6-bis[2-(trifluoromethyl)benzylidene]cyclohexanone was synthesized by an Aldol condesation between two molecules of 2-(trifluoromethyl)benzaldehyde and cyclohexanone, which was synthesized according our earlier published method [1, 2]. An amount of 7.5 mmol cyclohexanone was added to a solution of 15 mmol 2-(tri- fluoromethyl)benzaldehyde in MeOH (10 ml). The solution was stirred at room temperature for 20 min, followed by dropwise addition of NaOMe/MeOH (1.5 ml, 7.5 mmol). The mixture was stirred at room temperature and monitored by *TLC*. When the reaction was finished, the residue was poured into saturated NH_4Cl solution and filtered. The precipitate was washed with water and cold ethanol, and dried in vacuum. The solid was purified by chromatography over silica gel using CH_2Cl_2/CH_3OH as the eluent to yield compound. All chemicals used for the synthesis were commercially available of AR grade, and were used as received. The yellow crystal of the title compound was obtained by slow evaporation of a CH_3OH solution at room temperature.

Experimental details

Position of the H atoms were calculated based on geometric criteria (C–H = 0.97 and 0.93 Å for methyl and aromatic atoms, respectively) then were placed in their calculated position and refined isotropically using a riding model with U_{iso}(H) = 1.5 U_{eq}(C) for methyl and U_{iso}(H) = 1.2 U_{eq}(C) for all others.

Discussion

Curcumin has been demonstrated to possess a wide range of pharmaceutical activities, such as anti-tumor [3, 4], anti-inflammation [5], anti-oxidation [6], anti-bacterial [7], and cardiovascular protection [8, 9]. However, its high metabolic instability and low bioavailability have dramatically limited its practical application [10]. Recently, our research group has synthesized a series of mono-carbonyl analogs of curcumin (MACs) by deleting β-diketone moiety [11–13]. The title compound, belongs to MACs and shown greater metabolic stability than that of curcumin. The crystal structure of the title compound is depicted in the figure. The asymmetric unit of this complex is constructed by two 2-(trifluoromethyl)benzylidene groups and a cyclohexanone linker. The C–C bond length of the cyclohexanone moiety ranging from 1.491 to 1.509 Å are quite normal single bonds. The C7–C8 and C12–C14 bond lengths of 1.328 and 1.324 Å conform to the value for a double bond. The C6–C7 and C14–C15 bond length of 1.466 Å are intermediate between the double and single bonds. The shortening of the C–C bonds showing the partial double-bond character of the C–C bonds, which are influenced by adjacent conjugated double bonds.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.3817	0.2658	0.8542	0.118
H(3)	4*e*	0.3059	0.1607	0.9532	0.138
H(4)	4*e*	0.1948	0.0322	0.9011	0.123
H(5)	4*e*	0.1569	0.0069	0.7505	0.097
H(7)	4*e*	0.1698	0.1563	0.5789	0.073
H(9A)	4*e*	0.3648	–0.0376	0.6524	0.086
H(9B)	4*e*	0.2043	–0.0852	0.6257	0.086
H(10A)	4*e*	0.4067	–0.1602	0.5615	0.088
H(10B)	4*e*	0.4664	–0.0738	0.5165	0.088
H(11A)	4*e*	0.3190	–0.1581	0.4105	0.082
H(11B)	4*e*	0.1719	–0.1554	0.4708	0.082
H(14)	4*e*	0.1688	0.0338	0.3034	0.077
H(16)	4*e*	0.1185	–0.1913	0.3063	0.107
H(17)	4*e*	0.1400	–0.2884	0.1907	0.128
H(18)	4*e*	0.2399	–0.2454	0.0591	0.143
H(19)	4*e*	0.3256	–0.1030	0.0422	0.124

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1)	4*e*	0.4249(3)	0.3394(1)	0.7209(1)	0.199(2)	0.084(1)	0.122(2)	–0.038(1)	0.022(1)	–0.033(1)
F(2)	4*e*	0.2182(3)	0.3102(1)	0.6393(1)	0.141(2)	0.083(1)	0.125(2)	0.039(1)	0.025(1)	0.028(1)
F(3)	4*e*	0.4333(2)	0.2488(1)	0.6130(1)	0.122(1)	0.079(1)	0.105(1)	–0.0059(9)	0.037(1)	–0.0136(8)
F(4)	4*e*	0.4295(2)	0.0611(1)	0.2191(1)	0.128(1)	0.112(1)	0.079(1)	–0.019(1)	0.005(1)	0.0106(9)
F(5)	4*e*	0.2169(3)	0.0887(1)	0.1442(1)	0.141(2)	0.097(1)	0.126(2)	0.038(1)	0.004(1)	0.024(1)
F(6)	4*e*	0.4145(3)	0.0402(1)	0.0782(1)	0.184(2)	0.136(2)	0.076(1)	0.011(1)	0.047(1)	0.018(1)
O(1)	4*e*	0.1016(2)	0.1018(1)	0.4367(1)	0.096(1)	0.0602(9)	0.061(1)	0.0131(8)	–0.0049(8)	0.0045(7)
C(1)	4*e*	0.3078(3)	0.2021(2)	0.7413(2)	0.090(2)	0.062(1)	0.064(2)	0.018(1)	0.002(1)	–0.009(1)
C(2)	4*e*	0.3339(4)	0.2142(2)	0.8327(2)	0.143(3)	0.076(2)	0.075(2)	0.023(2)	–0.011(2)	–0.019(2)
C(3)	4*e*	0.2904(5)	0.1514(2)	0.8917(2)	0.188(4)	0.094(2)	0.060(2)	0.038(2)	–0.006(2)	–0.004(2)
C(4)	4*e*	0.2243(5)	0.0749(2)	0.8606(2)	0.152(3)	0.094(2)	0.062(2)	0.026(2)	0.013(2)	0.017(2)
C(5)	4*e*	0.2008(3)	0.0601(2)	0.7703(2)	0.101(2)	0.076(2)	0.066(2)	0.009(1)	0.006(1)	0.008(1)
C(6)	4*e*	0.2408(3)	0.1224(2)	0.7080(2)	0.071(1)	0.064(1)	0.059(1)	0.012(1)	0.004(1)	0.002(1)
C(7)	4*e*	0.2071(3)	0.1077(1)	0.6116(1)	0.065(1)	0.060(1)	0.059(1)	0.001(1)	0.0071(9)	0.007(1)
C(8)	4*e*	0.2231(2)	0.0341(1)	0.5650(1)	0.055(1)	0.055(1)	0.057(1)	–0.0034(9)	0.0049(9)	0.0042(9)
C(9)	4*e*	0.2900(3)	–0.0508(2)	0.6025(2)	0.083(2)	0.064(1)	0.065(1)	0.005(1)	–0.009(1)	0.007(1)
C(10)	4*e*	0.3727(3)	–0.1048(2)	0.5346(2)	0.072(1)	0.065(1)	0.084(2)	0.012(1)	0.002(1)	0.010(1)
C(11)	4*e*	0.2637(3)	–0.1225(2)	0.4529(2)	0.081(2)	0.058(1)	0.066(1)	0.009(1)	0.009(1)	0.002(1)
C(12)	4*e*	0.2092(2)	–0.0381(1)	0.4081(1)	0.057(1)	0.058(1)	0.056(1)	0.0004(9)	0.0047(9)	0.0016(9)
C(13)	4*e*	0.1731(2)	0.0378(1)	0.4675(1)	0.056(1)	0.054(1)	0.058(1)	–0.0030(9)	0.0066(9)	0.0066(9)
C(14)	4*e*	0.1949(3)	–0.0234(2)	0.3205(2)	0.069(1)	0.061(1)	0.062(1)	0.004(1)	0.003(1)	–0.000(1)
C(15)	4*e*	0.2149(3)	–0.0859(2)	0.2470(2)	0.077(2)	0.070(1)	0.061(1)	0.013(1)	–0.005(1)	–0.004(1)
C(16)	4*e*	0.1623(4)	–0.1726(2)	0.2536(2)	0.108(2)	0.076(2)	0.082(2)	0.002(2)	–0.004(2)	–0.009(1)
C(17)	4*e*	0.1739(5)	–0.2307(2)	0.1840(2)	0.151(3)	0.075(2)	0.090(2)	0.011(2)	–0.017(2)	–0.017(2)
C(18)	4*e*	0.2336(5)	–0.2055(2)	0.1061(3)	0.174(3)	0.093(2)	0.088(2)	0.030(2)	–0.013(2)	–0.033(2)
C(19)	4*e*	0.2851(4)	–0.1205(2)	0.0962(2)	0.139(3)	0.110(2)	0.061(2)	0.034(2)	0.004(2)	–0.012(2)
C(20)	4*e*	0.2772(3)	–0.0605(2)	0.1658(2)	0.091(2)	0.081(2)	0.056(1)	0.022(1)	–0.003(1)	–0.003(1)
C(21)	4*e*	0.3337(4)	0.0311(2)	0.1520(2)	0.112(2)	0.095(2)	0.054(2)	0.019(2)	0.006(1)	0.008(1)
C(22)	4*e*	0.3472(4)	0.2738(2)	0.6798(2)	0.105(2)	0.062(1)	0.084(2)	0.010(1)	0.016(2)	–0.019(1)

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